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Evolution of Martensitic Microstructure of Carbon Steel Tempered at Low Temperatures

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Abstract

This work presents the microstructural changes of a carbon steel during tempering at low temperatures (first stage of tempering), and its effects in the second and third stages. These changes are determined by internal friction. Internal friction is measured in a forced vibration mode at 5 Hz, in a temperature range from 300 to 600K. Four internal friction peaks appear: S1 at 350K, P1 at 380K, P2 at 440K, P3 at 480K. S1 only appears in the sample tempered at 380K for 20 hours, and this can be related to the reduction of the martensite tetragonality. The height of P1 is lowest when the intensity of tempering is increased. This height can be related to the quantity of epsilon carbide than precipitate. The heights of P2 and P3 are not changed significantly, but the difference of temperature between both is lowest when the tempering intensity is increased. This suggests than the dissolution of epsilon carbide and formation of cementite takes places in a lowest range of temperature.

Keywords: steel, quenching, tempering, internal friction

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1. Introduction

The strengthening mechanisms of carbon steel with martensitic microstructures tempered at low temperatures involve effects of the tetragonal distortion, mobility of carbon atoms, dislocation density and the interaction between dislocations and other defects. The understanding of these phenomena is important for a better understanding of the deformation mechanism and to design heat treatments for specific applications.

In other works [Hoyos et al., 2009; Hoyos et al., 2011], the internal friction was measured in two steels containing 0.626 and 0.71%wt C. The steels were quenched from 1093K, and tempered at 423, 573 and 723K, with tempering times of 10 and 60 minutes. Five internal friction were identified: P1 at 215K, P2 at 235K, P3

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at 260K, P4 at 380K and P5 at 420K, for 3 Hz. P1 and P2 were attributed to the interaction of dislocations with carbon atoms and carbides, respectively. P3 was attributed to the kink pairs formation on dislocations. P4 was related to the precipitation of epsilon carbide, and P5 to the motion of dislocations.

In previous work [Hoyos et al., 2012], the effects of tempering temperature and time obtained during the first stage of tempering on internal friction of martensitic carbon steel were presented. The steel was quenched into water and tempered at 340, 380, 420 and 460K for 10 min, and tempered at 380K for 1 and 20 hours. The internal friction was measured as a function of temperature from 300 to 600K, at 3 Hz.

The microstructural characterization appears in previous work [Hoyos et al., 2012]. In quenched sample, the microstructure is composed of martensite and austenite. Three stages could be distinguished during tempering. First stage is characterized by the precipitation of epsilon carbide, and it takes place between 350 and 420K. Second stage is characterized by transformation of retained austenite between 480 and 510K. In third stage, cementite precipitation occurs between 510 and 600K. Stages II and III are similar in all samples. However, when the tempering temperature of the samples is increased the stage I is disappeared.

In this work, we studied the evolution of the microstructure during the first stage of tempering, and its effects in the subsequent stages using internal friction. The internal friction was measured at 5 Hz, from 300 to 600K. The samples show four peaks at 350, 380, 420 and 480K. The internal friction background is highest when the frequency is increased, and a new peak S1 appears.

2. Experimental

The carbon steel is cut in sheets with dimensions of 2.5 mm x 0.5 mm x 15 mm. Table 1 presents the chemical composition of steel. The chemical elements were determined by optical spectroscopy. The samples were heated at 1093K for 5 minutes, and rapidly cooled into water. After quenching, the samples were tempered at 340, 380, 420 and 460K for 10 minutes. Additionally, other samples were tempered at 380K for 1 and 20 hours.

Table 1. Chemical composition of carbon steel

Element	C	Si	Mn	Cr	Ni	S	P	Mo	Cu	Fe
wt%	0,709	0,209	0,685	0,294	0,037	0,005	0,019	0.016	0,009	-------

Dilatometry measurements were performed from 300 to 1000K, using heating rates of 0.8 and 2 K/min. Internal friction spectrum was measured in a torsion pendulum by forced vibrations method at 5 Hz, from 300 to 600K, with an amplitude deformation of $3x10^{-6}$ and heating rate of 0.8K/min. The internal friction spectrum has been deconvoluted into Debye peaks and a thermal background.

3. Results and discussion

Figure 1 shows the dilatometry curves for the quenched sample, with heating rates of 0.8 and 2 K/min. Three stages are observed during heating: (I) epsilon carbide precipitation, (II) austenite transformation, and (III) cementite precipitation. These stages appear due to the tempering of microstructure during heating. The temperatures of different stages are higher when the heating rate is increased.

Figure 2 shows the position of the inflection points for the quenched sample heating at 0.8K/min. The position of the inflection points is determined by the first derivate of the length change as a function of

temperature (right vertical axis). This length change represents the deviation from the ideal thermal expansion due to the tempering (left vertical axis).

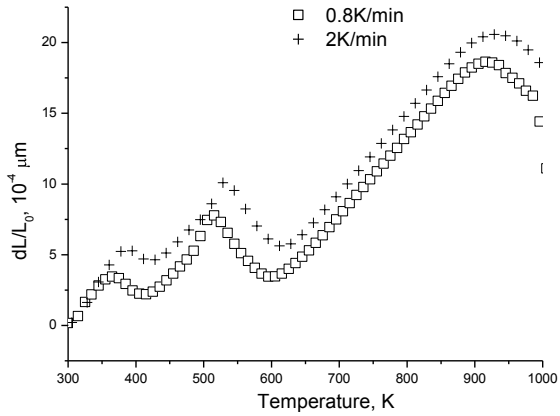

Fig. 1. Dilatomety curves of the quenched sample at two heating rates.

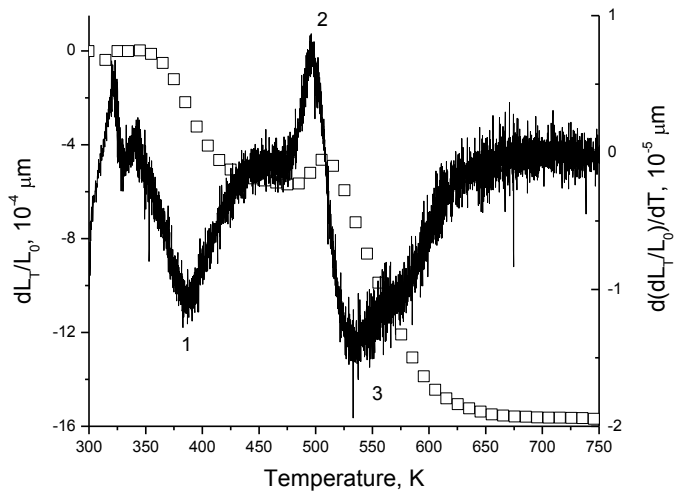

Fig. 2. Determination of the temperatures of tempering stages of quenched sample.

The activation energy can be derived from the slope of the straight line obtained by plotting ln (Ti^2/Φ) vs. l/Ti for different values of Φ (equation 1), where Q is the activation energy, Ti is the temperature (K) corresponding to the inflection point, ko is a constant, R is the gas constant and Φ is heating rate (K/min) [Waterschoot et al., 2006].

$$\ln\left(\frac{T_i^2}{\Phi}\right) \approx \frac{Q}{RT_i} + \ln\left(\frac{Q}{Rk_o}\right) \qquad (1)$$

Figure 3 shows the activation energy of stage I for all samples. It increases as the intensity of tempering is increased. The precipitation of epsilon carbide also increases with higher intensity of tempering. Thus, the number of carbon atoms in solid solution diminishes, and the new precipitation that takes place during dilatometry measurements is reduced, and it requires higher activation energy. The activation energies of stages II and III were similar for all samples. This suggests than these stages are not affected by tempering at low temperatures.

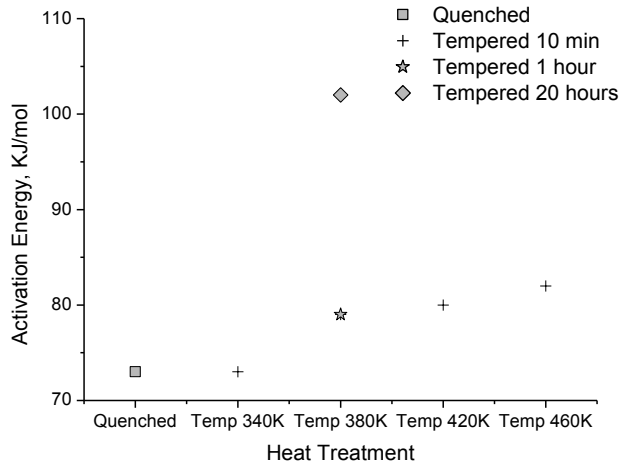

Fig. 3. Activation energies of stage I for all samples.

Figure 4 presents the internal friction spectrum of one quenched sample during heating at 5Hz, and another quenched sample during two successive heating cycles at a frequency of 3 Hz, and the measurements of the relative modulus during first heating at 3Hz. Three peaks appear: P1 at 380K and M1 at about 480K. Since M1 is broader than a Debye peak, and it is considered as a superposition of two Debye peaks: P2 at 440K and P3 at 480K. During second heating at 3 Hz, P1 and P2 disappear due to the tempering effects of the first heating.

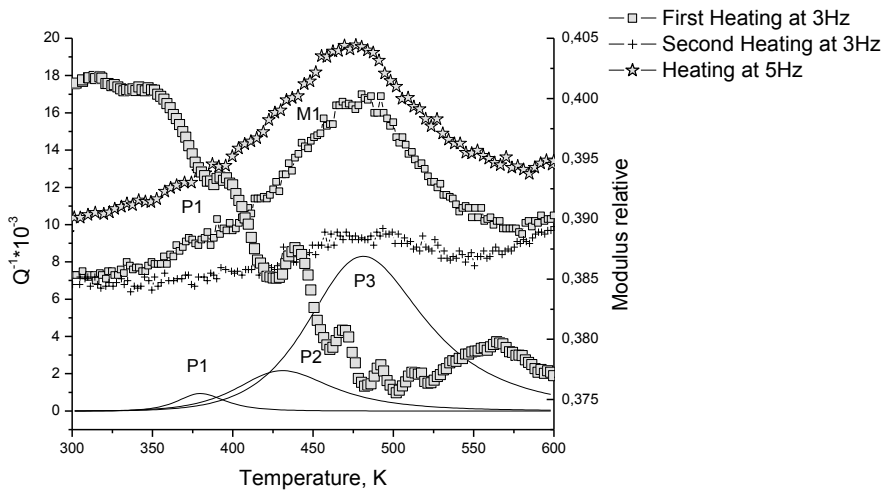

Fig. 4. Internal friction spectrum of quenched sample.

In relative modulus, different steps appear at temperatures of internal friction peaks. This confirms than the peaks are associated to the relaxation mechanisms. The internal friction background is highest when the frequency is increased, but the peaks appear at similar temperatures. Higher frequency measurements are required for the evaluation of activation energies.

Figure 5 shows the internal friction spectrum of the samples tempered for 10 minutes at different temperatures, measured at 5 Hz. Figure 6 presents the internal friction spectrum of the samples tempered at 380K for different tempering times, at 5 Hz. In all samples, peaks P1, P2 and P3 appear. Additionally, in the sample tempered at 380K, measured at 5 Hz, the peak S1 at 350K appears. This one is related to the Snoek effect, and its presence can be associated to the reduction of the martensite tetragonality.

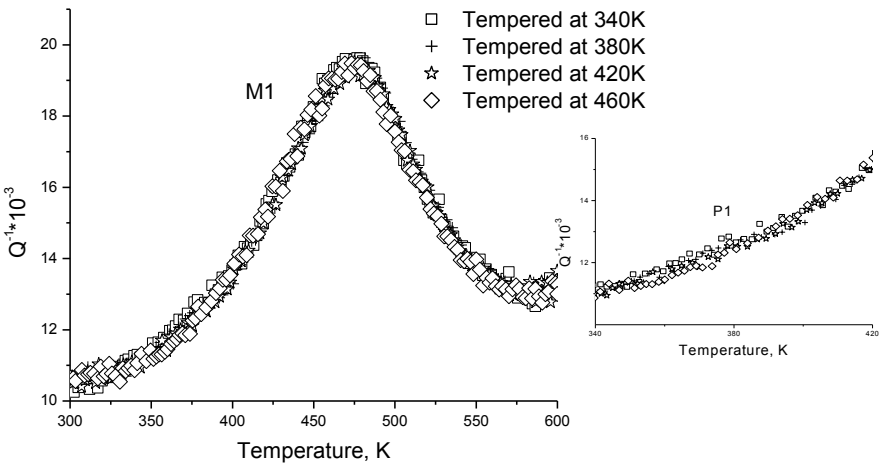

Fig. 5. Internal friction spectrum of samples tempered for 10 minutes.

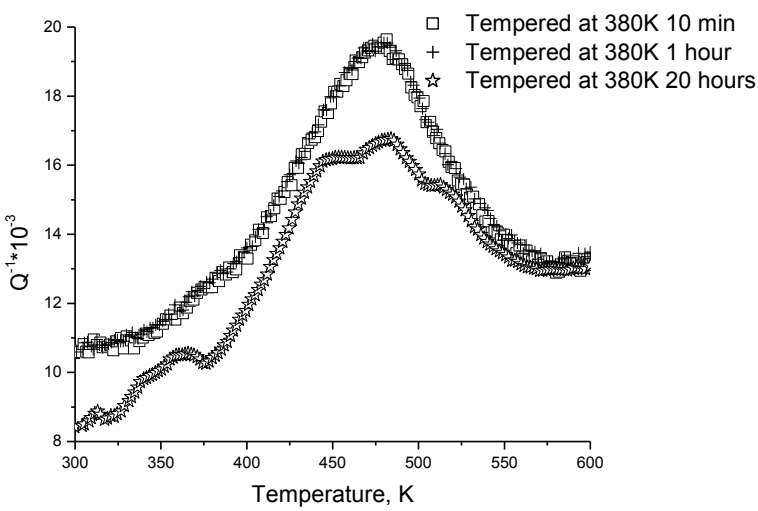

Fig. 6. Internal friction spectrum of samples tempered at 380K.

The height of P1 is lowest when the intensity of tempering is increased. This is an indication that this peak can be related to the precipitation of epsilon carbide. This precipitation is higher when the intensity of tempering is increased. Thus, the precipitation during the measurements of internal friction is lower. This explanation is supported by the results obtained in the two successive heating processes of quenched sample. P1 appears during the first heating and it disappears during the second heating. It can be interpreted by a relaxation mechanism due to the direct action of the precipitates on the substructure. According to Tkalcec [Tkalcec et al. 2006], this peak is not thermally activated and can be interpreted by a relaxation mechanism due to the interaction of dislocations and carbon content in solid solution. In both cases, this peak can be used for the evaluation of the precipitation of epsilon carbide.

P2 and P3 can be associated to the interaction of the thermally activated dislocations with epsilon carbide and cementite, respectively. The superposition of these peaks results in the formation of M1. M1 is asymmetrical and much broader than a single Debye peak due to the dynamic of precipitation. As it is know, the transition of epsilon carbide to cementite is preceded by the dissolution of epsilon carbide. Thus, the interaction of dislocations with epsilon carbide and cementite results in an asymmetrical and broad peak. The difference of temperatures between peaks P2 and P3 is highest when the tempering intensity is increased. This suggests than the transition of epsilon carbide to cementite is easier when the previous tempering intensity is lowest.

4. Conclusions

The precipitation of epsilon carbide is highest when the intensity of tempering is increased during tempering at low temperatures. This increases the activation energy required for the new precipitation of epsilon carbide during second heating, but it has no significant effect in the transformation of retained austenite and the precipitation of cementite.

Four internal friction are identified: S1 at 350K, P1 at 380K, P2 at 440K and P3 at 480K. S1 only appears in the sample tempered at 380K for 20 hours and it is attributed to the Snoek effect. P1 is related to the precipitation of epsilon carbide, and its height is lowest when the intensity of tempering is increased. P2 and P3 are related to the interaction of dislocations with epsilon carbide and cementite, respectively. Internal friction can be used to study the dynamics of the transition of epsilon carbide to cementite. But, measurements at higher frequencies are required for the evaluation of activation energies.

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